TENDER ENERGY X-RAY ABSORPTION SPECTROSCOPY (TES)

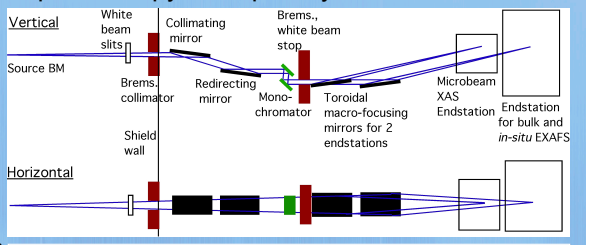
Project Team: S. Bare[1,2], J. Brandes[3], T. Buonassisi[4], J. Chen[5,2], M. Croft[6], E. DiMasi[7], A. Frenkel[8,2], D. Hesterberg[9], S. Hulbert[7,2], S. Khalid[7], S. Myneni[10], **P. Northrup[7,11]**, E.T. Rasbury[11], B. Ravel[12], R. Reeder[11], J. Rodriguez[7,2], D. Sparks[5,13], V. Stojanoff[7], G. Waychunas[14]

[1]UOP LLC, [2]Synchrotron Catalysis Consortium, [3]Skidaway Inst. of Oceanography, [4]MIT Laboratory for Photovoltaics Research, [5]Univ. of Delaware, [6]Rutgers Univ., [7]Brookhaven National Lab, [8]Yeshiva Univ., [9]North Carolina State Univ., [10]Princeton Univ., [11]Stony Brook Univ., [12]NIST, [13]Delaware Environmental Inst., [14]Lawrence Berkeley National Lab

NATIONAL SYNCHROTRON LIGHT SOURCE II planned by Photon Sciences Directorate

TECHNIQUES: High performance and *in-situ* X-ray absorption spectroscopy and spatially-resolved XAS of structured materials; optimized for the "tender" energy range from 1.2 up to 8 keV.

- Will enable **spatially-resolved** and ***in-situ*** studies of speciation and local structure by XAS, X-ray fluorescence (XRF) and spectroscopic imaging, in a *non-vacuum environment.*
- Chemical sensitivity to **key lighter elements** Mg through Ti, and advantageous heavier-element L and M edges such as Cd, Pd, U.
- Optimized for the **NSLS-II dipole bend** source: high brightness over a tunable spatial resolution and energy scanning across 1.2-8 keV.



CAPABILITIES:

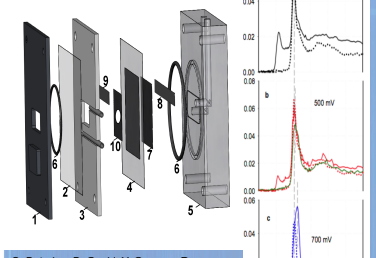
Source: dipole bend magnet, E_c 2.39 keV. Two optimized endstations.
Energy Range: 1.2 to 8 keV (optimized for 1.2-5 keV).
Spatial Resolution: 1x1 mm to <1x1 μm; **Flux**: up to $3x10^{12}$ ph/sec.
Detection: high- and low-countrate XRF from 0.9 to 8.3 keV.
Speed: on-the-fly scanning for ~1 minute EXAFS and/or rapid imaging.

	High-performance and *in-situ* XAS endstation	**Microbeam XAS and spectroscopic imaging endstation**		
Energy (keV)	Flux, at 0.2x0.2 to 1.2x0.5 mm (ph/s at sample)	Flux, at 19x23 microns	Flux, at 6x7 microns	Flux, at 1x1 micron
1.2	$1.15 x10^{12}$	$5.4 x10^{11}$	$2.85 x10^{11}$	$6.8 x10^{9}$
2	$3.5 x10^{12}$	$2.3 x10^{12}$	$1.0 x10^{12}$	$2.4 x10^{10}$
3	$2.8 x10^{12}$	$1.9 x10^{12}$	$7.7 x10^{11}$	$1.8 x10^{10}$
4	$1.9 x10^{12}$	$1.2 x10^{12}$	$4.5 x10^{11}$	$1.1 x10^{10}$
5	$1.3 x10^{12}$	$7.2 x10^{11}$	$2.7 x10^{11}$	$6.4 x10^{9}$
7.5	$5.1 x10^{11}$	$2.15 x10^{11}$	$8.1 x10^{10}$	$1.9 x10^{9}$

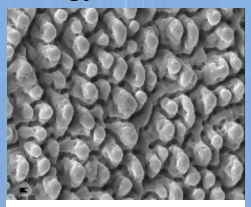
APPLICATIONS: Focus on *Energy*, *Climate*, *Soil* and *Earth* Sciences

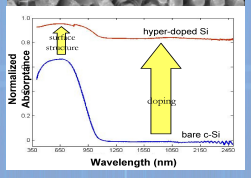
Sulfur poisoning of Fuel-Cell catalysts: *in-situ* XAS

• Novel **Spectroelectrochemical cell** design for *in-situ* measurements of fuel-cell catalyst at controlled electrochemical potential and under gas flow, all compatible with low-energy fluorescence-mode XAS measurements.
• Real fuel-cell materials and geometry.
• Sample spectra under varying potentials, indicating changes in S speciation and oxidation state.



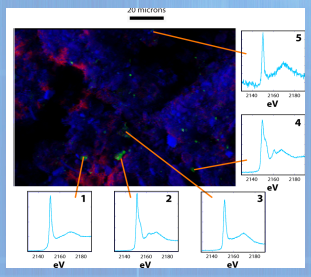
O. Baturina, B. Gould, Y. Garsany, R. Stroman and P. Northrup, "Identification of adsorbed SO2 species on fuel cell electrocatalysts by sulfur K-edge XANES combined with electrochemistry" in review.





Sulfur-doped silicon: Enhanced efficiency photovoltaics.

Surface structure (SEM) image of micro-textured and hyper-doped Si. Surface texture increases intrinsic absorption of the Si at the usual wavelengths; hyper-doping dramatically increases absorption at higher wavelengths.
B. Newman, T. Buonassisi, P.Northrup, in prep

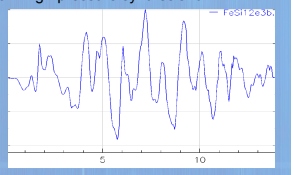


Phosphorus distribution and speciation: Key nutrient cycling and bioavailability influence global photosynthesis and biofuel/agricultural productivity.

Spatial and species heterogeneity of phosphorus in a natural sediment. Colors represent P (green), Si (blue) and Na (red). Point spectra indicate a variety of species.
Ingall, Brandes, Diaz, deJonge, Paterson, McNulty, Elliott, Northrup, "Phosphorus K-edge XANES spectroscopy of mineral standards," J. Synch. Rad., 18 (2011).
JDiaz, Ingall, Benitez-Nelson, Paterson, de Jonge, McNulty, Brandes, "Marine Polyphosphate: A Key Player in Geologic Phosphorus Sequestration" Science, 320, (2008).

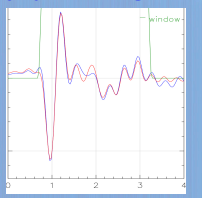
Investigation of site disorder in high-pressure silicate phases: Si K-edge EXAFS.

EXAFS data (k^2-weighted) suitable for determination of site disorder in microgram-size sample from high-pressure synthesis run.
P.Northrup, L. Li, D. Weidner, in prep



Nutrient sorption processes in poor and highly-leached soils: P, S, Ca, Mg, and K EXAFS.

S K-edge EXAFS (Fourier transform, real) data and fit for sulfate adsorbed to gibbsite mineral surface. Fit includes S-O, multiple scattering, and distances to two Al and one O in the substrate.
P. Northrup, M. Alves (USP, Brazil), D. Sparks, in prep.



SCIENTIFIC IMPACT:

- Energy Materials: Photovoltaic, fuel-cell, battery and superconducting (nano)materials.
- Catalysis/Chemistry: Materials (zeolites, thin films, nanomaterials), reaction mechanisms and intermediate species, poisoning of catalysts.
- Environmental/Earth Science: Biogeochemical and redox processes, contaminant behavior and remediation; Ca-Mg-Si high-pressure phases.
- Climate: Terrestrial/marine C cycling, carbonate (bio) mineralization, geologic record of climate change, ocean chemistry, CO_2 sequestration.
- Sustainability: Nutrient cycling, transport and bioavailability, biofuel/biomass productivity, especially in poor and leached tropical soils.

DEVELOPMENT STRATEGY:

- Our goal is *high productivity at the earliest possible date*.
- Strategy combines in-house and external aspects to create world-class capabilities and develop cutting-edge research programs -- and be ready to go on Day One.
- Utilize upgraded facilities at NSLS **X19A** Facility Beamline and **X15B** User Consortium-operated beamline.
- Design, commission TES microprobe endstation at X15B.
- Synchrotron Catalysis Consortium and BNL Chemistry: ongoing development of *in-situ* programs at X19A.
- Pre-test and commission optical components at NSLS.
- Collaborate closely with **ISS** (NEXT beamline for high-flux hard X-ray spectroscopies), **XFM** (hard X-ray micro-spectroscopy beamline), and **SRX** (Project Beamline: submicron hard X-ray probe) development teams.